NO DRAWINGS

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### COMPLETE SPECIFICATION

# Flavoring Material and process for Manufacturing same from Cocoa Shell-Containing, Chocolate Manufacturing **By-Products**

We, PETER PAUL, INC., a corporation organized under the Laws of the State of Delaware, United States of America, of Naugatuck, Connecticut, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which is to be performed, to be particularly described in and by the following statement:-

This invention relates to the production of a cocoa-like flavouring material from cocoa shell-containing, chocolate manufacturing byproducts and use of such materials in edible substances.

Cocoa beans in their shells are conventionally processed by roasting the same at elevated temperatures until the kernel dehydrates and shrinks away from the shell wall. The shells are then cracked and separated from the kernels which are further processed in various ways to produce cocoa powder and other known chocolate products. Those shell fractions containing a significant quantity of kernel or nibs are heated and pressed to re-25 move their fat content for various uses, the residue from this procedure being known as "expeller cake." The shells and expeller cake are normally considered waste by-products for which little use other than as a fertilizer has 30 been found.

It is primary object of this invention to provide a procedure for utilizing cocoa shellcontaining by-products in the manufacture in a simple and inexpensive means of a cocoalike flavouring material useful in edible foods for human consumption in lieu of conventional cocoa powder to provide exceptionally flavorable products. Further, the products hereof have no ingredients more harmful to the human body than cocoa powder itself and, in fact, are lower in fat content thereby making the same more desirable for use in various diet foods. Additionally, the extract hereof has fines fractions are normally further processed [Price 5s. 0d.]

good shelf life being capable of storage under various conditions for extended periods.

In addition to the basic concept of the instant invention, that is, the production of a cocoa-like flavoring material from normally waste by-products, various procedures are suggested herein for upgrading or improving the product realized, while still utilizing relatively simple manipulative steps and relatively inexpensive processing materials. To this end, it has been recognized that cocoa shells, like the cocoa beans themselves, vary in quality from batch to batch particularly in relation to the region in which the cocoa beans were grown. Thus, while with certain cocoa shells the most simple of the procedures disclosed herein may be adequate to provide the desired product, in other instances it may be of value to modify the procedures somewhat in order to increase the quality of the cocoa-like flavoring material obtained.

As pointed out hereinabove, cocoa shells heretofore have normally been considered as a waste by-product utilizable only as a fertilizer material. After cracking the shells to remove the same from the roasted cocoa beans, they are normally separated from the kernels by a conventional air classification procedure. Basically, the cracked shells can be separated into a coarse fraction, a medium fraction, and a fines fraction, the former comprising the major portion and having a particle size larger than a 6 mesh screen (U.S. Standard sieve), the intermediate fraction having a small percentage of nibs with the shell and generally passing through a 6 mesh screen and being received on a 16 mesh screen, and the latter fraction including a larger percentage of nibs although too much shell is present to be utilized in the manufacture of cocoa powder, this fraction being the remainder which passes through the 16 mesh screen. The medium and

resulting in the expeller cake. To appreciate the general form of these various materials, the following table sets forth analytical results of one sample of by-products comparing the same with conventional cocoa powder.

% Fat % Fiber % Protein Dry As Dry Dry As As Wgt. Rec'd Rec'd Wgt. Rec'd Wgt. Material Moisture 17.7 17.6 4.4 18.3 4.4 0.6 18.2 Coarse Shells 16.4 16.5 8.1 15.8 8.0 15.7 Medium Shells 0.8 11.1 20.9 11.0 20.7 17.6 Fines 1.0 17.4 22.4 5.1 21.8 20.6 21.2 5.0 Expeller Cake 2.8 4.3 13.6 4.2 26.7 13.3 Cocoa Powder 26.7 2.4

The above information is set forth as background to facilitate an understanding of the procedures to be defined hereinafter.

In its basic aspects, the present invention provides a flavoring material prepared by a process which comprises steeping a cocoa shell-containing material in an aqueous liquid at an elevated temperature for a period of time sufficient to effect substantial extraction of the flavoring material from the shells, and separating the aqueous liquid from the residue of said cocoa shell-containing material, after which the liquid is concentrated, if desired.

While it is obvious that the particular quantitative relationship between the cocoa shell-containing material and the water, as well as the processing temperature and time, may be varied over relatively large ranges, optimum extraction has been obtained by adding approximately 200 grams of cocoa shell-containing material for each liter of water and maintaining the mixture at a temperature of from about 95 to 100°C. for approximately 1 hour.

The use of various organic solvents in place of the water, while possible, has not been 30 found necessary in view of the desirable results obtained by an aqueous system which offers obvious advantages from the standpoint of economy and processing ease.

Vigorous stirring of the mixture of cocoa shell material and water was found to emulsify and remove a significant amount of the fat, about 11 percent of that normally present, although the quality of the extract was not otherwise improved. Extension of the extraction period substantially beyond one hour or use of multiple extractions have also been found to be of little value. Substantially all of the removable flavor is simply and effectively extracted by a single one-hour processing treatment in water maintained at about 95 to 100°C.

Surprisingly, it has been discovered that, while a cocoa-like flavoring material may be extracted from the fines, it is not particularly strong or tasteful as compared with extract produced utilizing medium or coarse shells. In fact, in contradistinction to what would normally be expected, the flavoring material obtained by extraction from the coarse shells was found to impart a better taste to edible products than either the extract of medium shells, fines, or even cocoa powder itself. Approximately twice as much fines-extract was necessary to impart a cocoa-like flavor in edible products comparable to a quantity of flavoring material extracted from coarse shells.

Thus, while a useful flavoring material may be produced from fines or medium shells, it has been found most desirable to utilize substantially all coarse shells in the procedure set forth hereinabove.

Preferably, the liquor obtained during the extraction procedure is separated from the residue by a suction filtration or other similar procedure. The residue is then washed with additional water and the filtrate and washings are mixed and subjected to a conventional vacuum evaporation procedure in order to concentrate the product approximately 5 times to form a syrup of sufficient strength for use in most applications.

While the simplest procedure, that is a hot-water extraction, has been shown to produce a cocoa-like flavoring material which is acceptable as a cocoa powder replacement, improvement in the flavor of the extract can be realized by various modifications of the basic processing treatment of the cocoa shell materials. For example, a susbtantial improvement can be effected by the incorporation of a small quantity of a mildly alkaline compound

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in the mixture of cocoa shell material and water during the extraction treatment. Exemplary of such additives are sodium carbonate, potassium carbonate, and ammonium carbonate, the most desirable compound being sodium carbonate in an amount equal to approximately 5 percent based on the weight of the shells

The addition of the carbonate raises the pH of the flavoring material to approximately 8. While this product may be used directly, it has been found advantageous to reduce the pH somewhat by addition of an acidic material. The pH of a flavoring material produced in the absence of the carbonate is approximately 5.2. While it is possible to reduce the pH of the carbone-treated material to the original level, it has been found to be desirable to merely render the material neutral, that is, lower the pH to approximately 7. Numerous acidic materials are useful for this purpose including phosphoric acid, sulfuric acid, and tartaric acid although best taste results having been realized with the use of phosphoric acid. Further, this material is particularly advan-25 tageous in that it is relatively inexpensive when considered in contrast to tartaric acid and may be readily obtained as a food grade acid.

Finally, with the use of certain shells, an improvement in the flavor of the product material has been effected by pretreating the cocoa shell-containing material with an enzymatic material. To a mixture of cocoa shellcontaining material and water preheated to approximately 60°C. with or without the carbonate, an effective quantity of the enzyme is added, the mixture being maintained at that temperature for approximately one hour before raising the temperature to the approximately 95 to 100°C. level utilized for the one-hour extraction procedure. The enzyme digestion is believed to break down the cell walls thereby facilitating the removal of the flavor constituents from the shells.

It should be noted that extraction of the flavor ingredients from cocoa shell allows the recovery of a residue representing 75 percent of the original shell weight. This residue is perfectly suitable for utilization as an organic soil builder and fertilizer.

The syrup obtained by the extraction of

shells and concentration of the extract as already generally described is comprised of about 25 percent dry matter. This dry matter is at least equivalent in flavoring strength to conventional cocoa powder. Thus it can be stated that 4 parts of the syrup referred to above is equivalent to 1 part of cocoa powder.

In addition to being substantially less expensive, various other advantages are realized through the use of this material. One major factor is the production of a lower fat cocoalike flavoring material. While various processes are known for the production of cocoa powder, none results in a product having a fat content substantially below about 10 percent by weight. As will be seen from the examples hereinafter, the fat content of the cocoa-like flavouring material of this invention is less than 1 percent. Thus, the extract provided hereby is more healthful in that it contains substantially less objectionable highly saturated fats such as are present in cocoa powder. Additionally, this material in reducing the fat content is comparatively low calorie in contrast with cocoa powder, a factor particularly desirable to those interested in reducing their weight level.

Having now described the instant inventive concept in a general manner, the following examples are set forth in order to more specifically illustrate the same, although it is to be understood that these examples are not to be considered in a limiting sense.

#### Example 1

Approximately 200 grams of by-product cocoa shells were soaked in one liter of water at 95 to 100°C. for approximately one hour. The liquid extract was filtered off using a suction technique and the residue was washed with about 300 milliliters of hot water. The filtrate and washings were concentrated approximately five times in a vacuum evaporator. This procedure was repeated utilizing (a) only coarse shells (b) only medium shells, and (c) only fines.

Taste tests were carried out by incorporating equivalent portions of various extracts into a standard drop cookie recipe. Control cookies were made from cocoa powder for comparative purposes. The recipe utilized was as follows:

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1 Cup brown sugar \( \frac{1}{2} \) Cup shortening 2 eggs 1½ Cup flour

½ teaspoon salt 1 teaspoon baking soda ☆ Cup milk 1 teaspoon vanilla

Shell extract syrup (eq. to \frac{1}{2} Cup cocoa powder)

Cream shortening and sugar together. Add vanilla. Add beaten eggs and continue to beat until fluffy. Sift flour and add to it baking soda and salt and sift again. Add dry ingredi-

ents alternately with milk. Beat until blended. Drop by teaspoonfuls onto a greased baking sheet. Space 2 to 3 inches apart. Bake at 370° for 10 to 12 minutes.

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Most persons on the cookie taste panel found that the coarse shell extract yielded the strongest most desirable fiavor while the fines extract produced a weaker flavor. Medium shell extract was intermediate between the two. It was determined that approximately one-half of the quantity of coarse shell extract was sufficient to impart a satisfactory cocoa-like flavor to cookies as compared with the fines extract. Cocoa powder itself gave a weaker flavor which was found inferior to the coarse shell extract by the taste test panel.

#### Example 2

The process of Example 1 was repeated utilizing only the coarse shells flaked to less than 1 centimeter in size and including in the water bath various quantities of mildly basic materials as follows:

A-5.3 grams of potassium carbonate; resulting pH of filtrate 6.9;

B-10 grams of ammonium carbonate; resulting pH of filtrate 7.9;

C-5.3 grams of sodium carbonate; resulting pH of filtrate 7.4; and,

25 D—10 grams of sodium carbonate; resulting pH of filtrate 7.9.

The mixtures were then heated in an oven for one hour at about 100°C., filtered, the pH adjusted to about 5.0 with tartaric acid and concentrated to approximately 200 grams. The resulting extract of each sample was made into drop cookies as described in Example 1 along with control cookies utilizing an extract made without carbonate and further control 35 cookies utilizing cocoa powder. Each of the carbonate-containing cookies were preferred over the control cookies made with carbonatefree extract, the use of the carbonate removing a somewhat strong, slightly bitter after-taste 40 objected to by some members of the taste test panel with cookies formed of extract prepared without carbonate. While all of the carbonatecontaining extract cookies were found comparable in chocolate taste to the cocoa powder 45 cookies, cookies made utilizing Sample 2D, that is the 10 gram level of sodium carbonate were found most preferable by the panel. Although somewhat greater quantities of the sodium carbonate could be included in the extraction procedure, this is avoided because greater amounts have never shown superior performance and have in many cases shown poorer taste panel ratings. It is to be understood that shells differing insofar as variety, type of fermentation, etc. possess distinctly different requirements regarding amount of carbonate necessary.

## Example 3

As will be seen from Example 2, the pH of the filtrate is increased somewhat from an original level of approximately 5.2 by the use of the carbonate. Various tests were made wherein the pH of the final extract was lowered utilizing the following acidic materials:

A—Adjust to pH 5 with tartaric acid;
B—Adjust to pH 7 with tartaric acid;
C—Adjust to pH 7 with phosphoric acid;
D—Adjust to pH 5 with phosphoric acid;
E—Adjust to pH 6 with phosphoric acid;
F—Adjust to pH 7 with sulfuric acid; and
G—Adjust to pH 5 with sulfuric acid.

The results of a taste test panel showed definite preference for a pH 7 phosphoric acid adjustment and a control cookie made without adjustment. A comparative cost evaluation indicates that the expense of utilizing tartaric acid to adjust the pH is approximately 6 times as high as phosphoric acid and better than 50 times as expensive as sulfuric acid. In addition to the taste preference for phosphoric acid-adjusted extract, this material must be considered more attractive because of its wide use and acceptance as a food grade acid. It is noted that by merely lowering the pH to 7 rather than to its initial pH of approximately 5, the cost of neutralizing acid is reduced by a factor of greater than 3. Although the pH 7 phosphoric acid-adjusted extract and the "no adjustment" control were considered equally satisfactory by the taste test panel, it is preferred to adjust the pH at least to its neutral point because most formulations in which this flavoring would be used are probably at or near a neutral pH.

### Example 4

Having arrived at the best possible carbonate-extraction and acid-neutralization conditions from coarse cocoa shells, tests were made to determine the effect of various enzymatic digestions. A comparison was studied of cookies formed using extracts made according to the following procedures:

A—Formed by the procedure of Example 2D.

B—An enzyme control extract formed by adding 200 grams of shells and one liter of tap water preheated to 60°C. and held in an oven for one hour at that temperature with approximately 10 grams of sodium carbonate being added. This solution was placed in a boiling water bath until the internal temperature reached approximately 95°C. and the mixture was maintained at from 95 to 100°C. for one hour, filtered, washed with 300 milliliters of hot water, cooled, the pH adjusted to 7 with phosphoric acid, and concentrated to 200 grams.

C—The procedure of Example 4B was repeated except that 0.200 grams of Pectinol Ri0, and 0.200 grams of Gumase HP—150, enzyme materials manufactured by Rohm and Haas Company, were added to two separate mixtures prior to the 60°C, incubation period.

D-Two additional tests were run as in

Example 4C eliminating the sodium carbonate from the extracting mixture.

E-An additional sample was prepared utilizing the process of Example 4D but eliminating both the enzyme and the sodium

The results of comparison tests by a cookie taste test panel indicated that with certain shells th enzyme-digesting treatment produced a slightly improved flavor, while with other shells the carbonate control of Example 4A was found to be just as satisfactory. In all instances, comparative tests with cookies made from cocoa powder indicated a preference for the 15 extract cookies.

#### Example 5

Cocoa powder and the cocoa shell extract were analyzed simultaneously to determine whether or not the cocoa shell extract, when compared chemically with cocoa powder, would be expected to produce adverse physiological effects. Cocoa shell extract powder was prepared by extracting two batches of cocoa shells according to Example 2D and concentrating to 400 grams. The extract was then freezedried and 102 grams of extract solids recovered. It is pointed out that this procedure can be utilized, if desired, to produce a powdered extract, either free or spray drying the same, although for most purposes it has been found satisfatcory to merely concentrate the extract to a syrup. However, for the purpose of this test, a powdered material was prepared by grinding the extract solids, this powder and a cocoa powder being subjected to chemical analyses according to well known standard procedures.

Moisture - Moisture was determined by

subjecting weighed samples of coca powder and shell extract to a temperature of 110°C. for two hours in a forced draft oven. The percent moisture was then calculated from the loss in weight.

Ash — Ash was determined by igniting weighed samples in a muffle furnace for two hours at 5550°C. and calculating the percent ash from the loss in weight.

Crude Fat - Crude fat was determined by

a standard AOAC procedure.

Crude Fiber — Crude Fiber was determined by a standard AOAC procedure.

Crude Protein — Crude protein was determined by a standard micro-Kjeldahl procedure.

Alkaloids - Alkaloids were determined according to a procedure outlined by Moir and Hinks [Analyst, 60, 439 (1935)] for the analysis of foodstuffs containing cocoa material.

Tannins & Catechins — Tannins and

catechins were determined according to an AOAC procedure which has been modified slightly by Jensen [Analyst, 53, 365 (1928)] and Duthie [Analyst, 63, 27 (1938)].

Catechins — The filtrate from above was combined with 50 ml of Stiasny's reagent and boiled for one hour under an air condenser. The precipitate was filtered off, dried for two hours in a vacuum desiccator, and weighed to determine the amount of catechins present.

Total Reducing Substances, Total Reducing Sugars and Polyphenols -

The above three analyses were performed according to the procedures outlined in Technical Bulletin No. 1225, United States Department of Agriculture, entitled "The Chemical Composition of Representative Grades of the 1952 and 1954 Crops of Flue Cured Tobacco."

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0/0	Compos	ition
Dry	Weight	<b>Basis</b>

	Cocoa	Extract
Ash	5.9	19.6
Protein	24.3	9.7
Fat	14.3	0.7
Fiber	10.1	0.4
Reducing Substances (as Glucose)	5.6	8.0
Reducing Sugars (as Glucose)	2.8	0.6
Polyphenols (as Glucose by diff).	2.8	7.3
Alkaloids	1.6	2.6
Tannins	6.3	19.4
Catechins	1.8	12.0

It will be seen from the above that there are no materials present in the extract which might be expected to have any deletrious effect when used in food products for human consumption. In fact, the extremly low fat content will provide great advantages from the standpoint of eliminating cholesterol-forming highly saturated fats such as normally found in cocoa powder, and also in reducing the calorie content.

EXAMPLE 6

In order to test the storage stability of the extracts prepared according to the instant invention, bacteriological assays of these materials were made.

Three extracts of chocolate flavoring were carried out according to the procedure of Example 2D. The extraction products were mixed together and 50 g samples were taken for storage tests in eight sterilized jars. Three jars were placed in a freezer, five jars were placed in a refrigerator while four more 50 g samples were placed in jars and pressure-cooked for 15 minutes at 15 psi. These jar lids were then 25 tightened and stored at room temperature. The samples were removed from these test environments at seven-day intervals and bacteriological profiles were determined. The results of these bacteriological analyses appear in the following 30

Type of Sample	Storage Time	Plate Count	Yeast & Mold	Coliforms
Refrigerated	1 day	40,000	280	250
>>	7	30,000	N.S.*	N.S.
23	14	48,000	N.S.	N.S.
<b>33</b>	28	29,000	N.S.	N.S.
Frozen	7 days	40,000	N.S.	N.S.
22	14	37,000	N.S.	N.S.
<b>37</b>	28	35,000	N.S.	N.S.
Canned	1 day	N.S.	N.S.	N.S.
<b>33</b>	7	N.S.	N.S.	N.S.
33	14	6,500	550	N.S.
33	2.	N.S.	N.S.	N.S.

\*N.S. — Not Significant.

Example 7

Other edible products utilizing extract prepared according to the preferred process of Example 2D substituted for equivalent quantities of cocoa powder can be made according to the following procedures.

A. Chocolate Pudding 24 cup milk (Scald)

10 combine and stir until well blended

3 cup milk 4 cup cornstarch 2 tablespoons sugar

4 teaspoon salt

15 4-5 teaspoons shell extract syrup

Add these ingredients to the hot milk. Stir and cook them over a very low flame, or in a double boiler, until they thicken and the cornstarch is cooked (when you can no longer taste it). Beat until light:

1 egg 2 tablespoons sugar

Pour the hot mixture over the egg, beat it and return it to the heat for a minute or two. Stir it constantly until the egg thickens, then 25 remove it from the heat. When cool add:

½ teaspoon vanilla. Chill the pudding.

# B. Chocolate Flavored Frosting

111 lbs.	12 oz.	Shortening
78 lbs.	8 oz.	Skim Milk Powder
110 lbs.		Chocolate flavor extract
2 lbs.	13 oz.	Salt
1 lb.		Vanilla Flavor
	15 oz.	Lecithin

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Cream the shortening and chocolate flavor extract together in a commercial Hobart mixer and mix until creamy. Next add the lecithin and the remaining ingredients and mix until blended to a homogeneous syrup.

C. Chocolate Cake

2 cups brown sugar

1 cup shortening

2 eggs

 $\frac{1}{3}$  cup hot water with 2 teaspoons baking soda added.

2½ cups flour

1 cup buttermilk

1 teaspoon vanilla

15 1 teaspoon salt

> Cream shortening and sugar together, then add the vanilla and eggs and beat until fluffy. Next add the chocolate flavor extract and the hot water with baking soda. Finally add the flour and salt alternately with the buttermilk and mix until blended. Bake at 350° for 30-35 minutes.

D. Breakfast Cocoa

For each cup of cocoa add an equal amount

of the following ingredients:

1-2 tablespoons of chocolate flavor extract

1-2 tablespoons of sugar 1 cup of milk

Mix the chocolate flavor extract and sugar 30 together in a sauce pan. Add milk and bring to a near boil. Allow to simmer 5 minutes and serve.

Each of the above will produce an edible food as good or better than the same product

35 utilizing cocoa powder.

From the above detailed description it will now be seen that there is herein provided an improved flavoring material and method of manufacturing the same, for use particularly in edible food products for human consumption in lieu of cocoa powder.

## WHAT WE CLAIM IS:—

1. A process for preparing a flavouring material which comprises steeping a cocoa shell-containing material in an aqueous liquid at an elevated temperature and for a period of time sufficient to effect substantial extraction of the flavouring material and separating the aqueous liquid containing said flavouring material from the residue of said cocoa shellcontaining material.

2. A process according to claim 1, in which said cocoa shell-containing material consists essentially of cocoa shells which have been separated from cocoa beans after roasting the

beans with the shells thereon.

3. A process according to claim 2, in which said cocoa shell-containing material comprises coarse or medium grade cocoa shells or a mix-60 ture thereof.

4. A process according to claim 3, in which said shells are susbtantially all coarse cocoa shells.

5. A process according to claim 4, in which said shells are flaked to less than 1 cm.

6. A process according to claim 4 or claim 5, in which the ratio of said shells to the water in said liquid is about 1:5 by weight.

7. A process according to any one of claims 1 to 6, in which said temperature is from 95 to 100°C and said time is about 1 hour.

8. A process according to any one of claims 1 to 7, in which the aqueous liquid containing said flavouring material is concentrated by removing a portion of its water content.

9. A process according to claim 8, in which the concentration is effected by vacuum

evaporation.

10. A process according to claim 8 or claim 9, in which said residue is washed with hot water, the wash water is mixed with the aqueous liquid containing said flavouring material and the mixture of said wash water and said aqueous liquid containing said flavouring material is concentrated.

11. A process according to any one of the preceding claims, in which said aqueous liquid is separated from said residue by suction filtra-

tion.

12. A process according to any one of the preceding claims, in which a basic compound is added to said aqueous liquid during said steeping.

13. A process according to claim 12, in which said basic compound is sodium carbonate, potassium carbonate or ammonium

carbonate.

14. A process according to claim 13, in which said sodium carbonate is added in an amount of about 5% based on the weight of said cocoa shell-containing material.

15. A process according to any one of claims 12 to 14, in which an acid is added to said

aqueous liquid.

16. A process according to claim 15, in which sufficient acid is added to lower the pH of said aqueous liquid to about 7.

17. A process according to claim 15 or claim 16, in which said acid is phosphoric acid, 110 sulfuric acid or tartaric acid.

18. A process according to any one of the preceding claims, in which, prior to said steeping said cocoa shell-containing material is subjected to enzymatic digestion.

19. A process according to claim 18, in which said cocoa shell-containing material is added to said aqueous liquid at a temperature of about 60°C, an effective amount of an enzymatic material is added thereto to digest 120 said cocoa shell-containing material, said aqueous liquid is maintained at about 60°C for about 1 hour, the temperature of said aqueous liquid is subsequently elevated and the aqueous liquid is maintained at the elevated 125

temperature for a period of time sufficient to effect substantial extraction of the flavouring material.

20. A process according to claim 1, substantially as hereinbefore described with reference to the foregoing Examples.

21. A flavouring material when prepared by a process according to any one of the pre-

ceding claims.

22. A flavouring material according to claim 21, having a fat content of less than 10 weight percent.

23. An edible food product comprising a flavouring material according to claim 21 or claim 22, in association with conventional food 15 ingredients.

24. An edible food product according to claim 23, substantially as hereinbefore described with reference to the foregoing Examples.

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